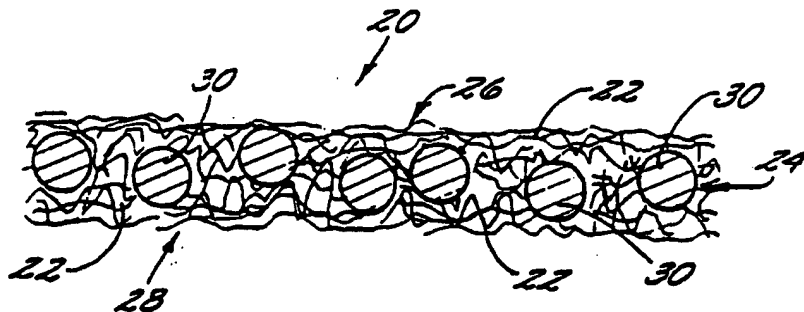




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<b>(21) International Application Number:</b> PCT/US93/11062 <b>(22) International Filing Date:</b> 15 November 1993 (15.11.93)  <b>(30) Priority data:</b> 07/977,995                      18 November 1992 (18.11.92) US  <b>(71) Applicant:</b> HOECHST CELANESE CORPORATION [US/US]; Route 202-206 North, Somerville, NJ 08876 (US).  <b>(72) Inventors:</b> GROEGER, H., Gunter ; 7716 Bradenton Drive, Charlotte, NC 28210 (US). SERAD, George, A. ; 3008 Cutchin Drive, Charlotte, NC 28210 (US). FELTON, Clinton, D. ; 3700 Barclay Downs Drive, Charlotte, NC 28209 (US).		<b>(74) Agent:</b> MCCANN, Philip, P.; Hoechst Celanese Corporation, P.O. Box 32414, Charlotte, NC 28232 (US).  <b>(81) Designated States:</b> JP, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.          Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>

**(54) Title:** FIBROUS STRUCTURE CONTAINING IMMOBILIZED PARTICULATE MATTER AND PROCESS THEREFOR

**(57) Abstract**

Fibrous structures containing immobilized particulate matter and processes for making them are disclosed. These structures may be used for filters and for fabrics including chemical defense garments and others. The fibrous structures have webs containing composite fibers having a lower melting component and a higher melting component. The particles are distributed into the interior of the web and fused to the low melting component of the individual fibers without substantially reducing the available surface area of the particle. A fibrous structure of a composite staple having a nylon sheath and a polyester core, with activated carbon particles bonded to the sheath is specifically disclosed. The processes for producing such a fibrous structure include carding a web of staple fibers and distributing particulate matter therein, air laying a web of staple fibers and distributing particulate matter therein, spin bonding a web of continuous filaments and distributing particulate matter therein, and wet forming a web of staple fibers and particulate from a suspension thereof between two wet formed layers of staple fibers. One or more additional layers of nonwoven, woven, or knit webs or fabrics may be bonded thereto.

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## FIBROUS STRUCTURE CONTAINING IMMOBILIZED PARTICULATE MATTER AND PROCESS THEREFOR

### Field of the Invention

The invention claimed herein relates to a fibrous structure having particulate matter contained therein. In particular, this invention relates to particulate containing filters and to chemical defense fabrics and other fibrous structures containing particulate matter.

### Background of the Invention

Particulate matter may be incorporated into a fibrous structure for a variety of reasons. For example, chemical defense fabrics for garments for military personnel may contain vapor sorptive particles such as activated carbon. These garments typically are designed to remove poisonous gases from the air. Filters may include sorptive particles selectively to remove certain components of a liquid or gas passing through the filter. Abrasive particles may be used to create an abrasive fabric. Particulate matter may also be used in a fibrous structure to release an active agent into a fluid stream.

Eian et al. U.S. Patent No. 4,868,032 discloses chemical protective garments. Reinforcing bi-component fibers are needled into a meltblown web of polymer fibers onto which solid particulate matter has been dispersed. Heat is applied to bond the needled fibers to the meltblown on both sides of the fabric.

Haruvy et al. U.S. Patent No. 4,872,220 discloses a protective clothing comprising a laminate of three layers in which one of the layers may contain an adsorptive particulate.

Giglia et al. U.S. Patent No. 4,904,343 relates to a toxic vapor adsorptive nonwoven fabric in which activated carbon fibers and activated carbon particles may be incorporated into a web of fibrillated acrylic fiber. The fabric is prepared by the wet forming process. A slurry of the particles and fibers is deposited onto a forming belt to form a particulate containing web that may be laminated to other fabrics.

As noted above, particulate containing structures typically are laminated to other fabric layers to produce garments. One problem that has been encountered is that garments that contain particulate matter typically do not last for a suitable period of time. Washing and other stresses can result in delamination of the fabric layers, loss of particulate matter, and deterioration of the garment. The particulate matter often is adhered to the surfaces of the particulate containing structure that is laminated to various fabrics to form the garment. The particles interfere with bonding between fabric layers, whether bonding is accomplished by heat, pressure, adhesives, or some combination of these methods. Also, the particles are subject to abrasive removal, which reduces the effectiveness of the garment.

Filters containing particulate matter typically are not laminated to fabric layers, it being desirable to avoid restricting flow of liquids or gases through the filter so as not to increase pressure drop across the filter. For example, Shimomai et al. U.S. Patent No. 3,998,988 discloses that filters may be prepared from finely divided adsorbent particles such as activated carbon adhered to a conjugate fiber of thermoplastic polymer composed of a higher melting component and a lower melting component. Fiber having a polyethylene core and a polystyrene sheath is specifically disclosed. The fiber is heated to bond the particles to the surface of the fiber without substantially adhering the fibers to one another. The fibers

may be in the form of a nonwoven web of filaments or staple fibers, or a knit, woven, or nonwoven fabric having particulate matter adhered to the surface thereof. U.S. Patent No. 3,998,988 does not recognize the problem of producing structures having particulate matter on the surfaces thereof and proposes no solution to the problem of having particulate matter on the surface of a web or fabric.

It would be desirable to produce open fibrous structures in which particulate matter may be immobilized within the fibrous structure between the surfaces thereof and does not interfere with bonding to other fibrous structures. It would also be desirable to produce a fibrous structure containing particulate matter that substantially is not subject to abrasion or abrasive removal of the particulate matter. Such open fibrous structures could be useful for filters and for laminating to fabrics for making garments or to other fibrous structures that do not as readily delaminate as prior garments or other structures.

#### Summary of the Invention

The invention claimed herein relates to a fibrous structure comprising a nonwoven web of manufactured fibers. Particulate matter is immobilized within the web without substantially extending into the upper and lower surfaces of the web.

The manufactured fibers may include thermoplastic manufactured fibers that can be thermally bonded to entrap the particulate matter within the web structure and to which the particulate matter can be fused. The thermally bonded web structure substantially limits migration of particulate matter within the structure or abrasive loss of particles from the structure. Composite thermoplastic fibers in which one component has a relatively lower

melting point and the other component has a relatively higher melting point may be used. The fibers are thermally bonded at the cross over points. The higher melting component provides strength to the fibrous structure while the particulate matter may be fused to the low melting component of the composite fiber.

In a specific embodiment, the fibrous structure comprises sheath-core composite fibers in which the sheath is a lower melting nylon and the core is a higher melting polyester. Activated carbon particles are entrapped within the thermally bonded fibrous structure without extending substantially into the surfaces of the structure and are fused to the nylon sheath component of the sheath-core composite fiber. The nylon sheath bonds the individual fibers at the cross over points.

In alternative specific embodiments, the nonwoven fibrous structure is a core structure and may have laminated thereto one or more fabric layers. These fabric layers may be nonwovens, woven fabrics, or knit fabrics. The core may include on one or both sides thereof partially densified nonwoven webs of composite fibers that are bonded to the core through the application of heat, or pressure, or a combination of both. This fibrous structure is also suitable for lamination with various fabric components or may be used as a filter.

The invention also relates to a process for producing the fibrous structure. The process comprises the steps of forming an open fibrous web of manufactured fibers that have particulate matter distributed within the web between the surfaces of the web without substantially extending into the web surface, immobilizing the particulate matter within the interior of the web, and thermally bonding the web. The web may be formed by forming

a carded web and distributing particulate matter therein; air forming a web and distributing particulate matter therein; wet forming a web having particulate matter distributed therein between two additional wet formed layers; or spinning of continuous filaments, forming an open web therefrom, and distributing particulate matter therein; or other methods in which a suitably open web receives particulate matter and entraps a particulate matter within the structure of the web. The particulate matter is immobilized within the web by applying heat to fuse the particulate matter to the fiber and to thermally bond the web.

In one specific embodiment, the particles are heated to a point above the melting point of the low melting component of the composite fiber and then distributed into the interior of the web where the heated particle causes localized meniscus bonding with the fibers. Thereafter, the web is thermally bonded.

In a still more specific embodiment, the open web containing the particulate matter is dry formed on a partially bonded web prior to dispersion of particulate matter into the open web. Thereafter heat is applied to fuse the particulate matter to the fibers of the open web, to thermally bond the web, and to bond the partially bonded web to the open web.

#### Brief Description of the Drawings

Figure 1 represents a fibrous structure comprising an open web of composite thermoplastic manufactured fibers having particulate matter immobilized therewithin in accordance with the invention.

Figure 2 represents the fibrous structure of Figure 1 having bonded thereto a partially densified web of manufactured fibers.

Figure 3 represents the fibrous structure of Figure 2 having bonded thereto opposite the partially densified web an additional open web of composite thermoplastic manufactured fibers with particulate matter immobilized therewithin and a partially densified web of manufactured fibers.

Figure 4 represents a cross section through a composite thermoplastic manufactured fiber as used to form the fibrous structure of the invention.

Figure 5 represents a particle entrapped within a portion of a fibrous structure and fusion bonding of the particle to the individual fibers.

Figure 6 represents thermal bonding of the fibrous structure of the present invention within which particulate matter is entrapped.

Figure 7 represents a specific embodiment for the manufacture of the fibrous structure of Figure 1.

Figure 8 represents a specific embodiment for the manufacture of the fibrous structure of Figure 2.



Figure 9 represents a specific embodiment for the manufacture of the fibrous structure of Figure 3.

Figure 10 is a highly schematic representation of a wet forming method for preparing a fibrous structure in accordance with the invention.

Figure 11 represents in longitudinal section a fibrous structure made by the wet forming method of Figure 10.

Corresponding reference characters indicate corresponding components throughout the several views of the drawings.

#### Detailed Description of the Preferred Embodiments

Figure 1 shows at 20 a greatly enlarged representation of a sectional view through the open fibrous structure of the invention. The fibrous structure comprises a plurality of individual thermoplastic composite manufactured fibers 22, the structure and composition of which are discussed in greater detail hereinbelow, which form an open web 24 and define an upper surface 26 and a lower surface 28 for the web. Particulate matter, represented at 30, is shown distributed throughout the web without substantially extending into the upper and lower surfaces of the web. The web 24 is sufficiently open so that particles 30 are distributed within the web between the surfaces 26 and 28. The web 24 is thermally bonded at the cross over points of the individual fibers 22, entrapping the particles so as substantially to preclude their migration out of the web (as illustrated greatly enlarged in Figure 6). The

particles 30 are fused to the individual fibers to secure the particles without substantially affecting the surface area of the particle available for adsorption (as illustrated greatly enlarged in Figure 5).

The open fibrous structure of the invention as shown in Figure 1 may be used in filter media. As set forth, one of the prime functions of the present invention is immobilizing the functional particulate matter, i.e. the carbon within that structure. Not only can immobilization of functional particulate matter be achieved, but also particulate filtration can also be achieved by using the fibrous structure of the present invention in a filter media. More specifically, it has been found that media containing the open fibrous structure in the present invention having 10-50 percent by weight of fiber and 50-90 percent by weight of particulate matter is suitable for both adsorption and particulate filtration. Preferably, the media contains 20-30 percent fiber by weight and 70-80 percent particulate matter. Such filter media is demonstrated adsorption and particulate removal efficiency, comparable to normal particulate filtration media.

Figure 2 shows at 32 a greatly enlarged representation of a sectional view of the fibrous structure of Figure 1 bonded to a partially densified web 34 at the interface 36 of the surfaces of the webs. Former surface 28 of web 24 is now bonded to web 34. Partially densified web 34 defines a lower surface 38 opposite interface 36. The partially densified web comprises composite thermoplastic manufactured fibers 40 that may be the same or different as the manufactured fibers 22, as explained further hereinbelow. Because it is partially densified, web 34 is now flattened and has fibers extending above its surface so that the fibers 22 and 40 intermingle to a degree that upon bonding, by application of heat or

pressure, or both, webs 24 and 34 form a unitary structure 32 that substantially precludes delamination at interface 36.

Figure 3 shows at 42 a greatly enlarged representation of a sectional view of the fibrous structure of Figure 2 having bonded thereto at interface 44 an open fibrous web 46 that contains particulate matter 48. Fibrous web 46 comprises composite thermoplastic manufactured fibers 50 that may be the same or different as the fibers 22 of web 24. Particulate matter 48 is entrapped within web 46 and fused to the individual fibers 50 in the same manner as with respect to web 24. Particulate matter 48 may be the same or different as particulate matter 30 of web 24.

Also referring to Figure 3, a partially densified web 52 is bonded to web 46 at interface 54 opposite interface 44. Partially densified web 52 defines an upper surface 56 opposite interface 54. The partially densified web comprises composite thermoplastic manufactured fibers 58 that may be the same or different as the manufactured fibers 50, 40, or 22, as explained further hereinbelow. Because it is partially densified, the surface of web 52 is not flattened and has fibers extending above the surface so that the fibers 50 and 58 are intermingled. Also, fibers 22 and 50 are intermingled at interface 44, and fibers 40 and 22 are intermingled at interface 36. Upon bonding of structure 42, by application of heat or pressure, or both, webs 34, 24, 46, and 52 form a unitary structure 42 that substantially precludes delamination at interfaces 36, 44, and 54.

Turning now to Figure 4 for a more detailed discussion of the composite thermoplastic manufactured fibers represented in Figures 1 through 3, Figure 4 is a

representation of a cross section through a composite thermoplastic manufactured fiber 60. Fiber 60 is illustrative of the many types of composite manufactured fibers that may be used in webs 52, 46, 24, and 34 of Figures 1, 2, and 3 and may comprise fibers 22, 40, 50, and 58. Composite manufactured fiber 60 is illustrated as a concentric sheath-core fiber in which the sheath and the core each comprise about 50% of the cross sectional area of the fiber. The fiber has a lower melting sheath 62 and a higher melting core 64. The sheath should have a melting point that is at least about 20°C below the melting point of the core and should occupy about half the cross section of the fiber to provide strong thermal bonding of the fibrous structure and of particulate matter to individual fibers without adversely affecting the integrity of the core. The core provides strength and integrity to the open fibrous structure. A range area of the fiber occupied by the sheath is contemplated to be from about 40% to about 60%. For example, in a preferred embodiment, the sheath may comprise a nylon that has a melting point of from about 175 to about 185°C. The core may comprise a polyester having a melting point of from about 240 to 256°C.

Concentric sheath-core fibers are one example of the composite thermoplastic manufactured fibers that can be used in the practice of the invention claimed herein. Suitable fibers include eccentric sheath-core fibers in which the center of the core does not coincide with the center of the fiber and side-by-side fibers in which two components, one higher melting and one lower melting, are simultaneously extruded through a single orifice to form a fiber having two halves. Composite fibers of these types are also referred to as by-component fibers or heterofil fibers.

The fibrous structures of the invention all include composite manufactured fibers as described above, having a lower melting component for thermal bonding of the structure and for fusing of particles to individual fibers. The skilled artisan will recognize that there are a wide variety of composite fibers having a higher melting component and a lower melting component that are suitable for the practice of the invention and that a nylon sheath and polyester core fiber is but one of the vast array of fibers available.

The composite fibers may be in a wide variety of forms including crimped and non-crimped cut staple fibers, short-cut staple, continuous filaments, or blends thereof. The composite fibers should be present in an amount sufficient to provide adequate bonding to individual particles and to form a structure that may be thermally bonded to entrap the particles. Additionally, the webs may include with the composite manufactured fibers, non-composite manufactured fibers, filament or staple, or other substances such as cellulose acetate fibrets. The fibrous structure of the invention may include from about 10% to about 80% by weight of non-composite fibers.

The composite fibers contemplated for use in the practice of the present invention include structural fibers having a denier per filament of from 1 to 15 to provide sufficient structure for entrapping particulate matter and for bonding thereto. A fabric structure including such structural fibers will also include microdenier fibers of from .1 to 1 denier per filament to provide softness for comfort in a garment. These microdenier fibers may be composite or non-composite manufactured fibers or a blend thereof. Fiber structures intended for use as filters will need to include, in addition to the structural fibers of from 1 to 15 denier per filament, structural fibers having a denier per filament of from 6 to 10,000

to provide stiffness, especially if a large particulate is used. Structural fibers of a denier per filament of from 6 to 10,000 to support relatively large particulate matter may be selected from composite fibers, non-composite fibers, and blends thereof. These fiber structures that are used for filters can be built to thicknesses of from 1/2 to 2 inches or more.

Turning now to a discussion of entrapping the particulate matter within the fibrous structure and fusing the particulate matter to the individual fibers, Figure 5 represents a single particle entrapped within a plurality of fibers 60. The fibers 60 are fused to the particle 30 at numerous points 66. Fusing of the particle to the fiber is localized in that the low melting component of a composite fiber does not form a film over the particle or otherwise substantially reduce the surface area of the particle available for contact with a liquid or gas stream moving through the fibrous structure.

Particulate matter 30 may be selected from a wide variety of substances having some function that is desirably incorporated into a fibrous structure. One of the most common is activated carbon adsorbent. Activated carbon beads may be fused to the individual fibers of the fibrous structure of the invention by heating the particles above the melting point of the low melting component of the composite thermoplastic fiber and dispersing the particles into the web or by heating the web after the particles have been dispersed therein. Activated carbon particles, and other inorganic oxides and hydrates have significantly lower specific heats than polymers and so heat quickly and result in localized fluidity of the low melting polymer component. A "spot weld" is produced with a thin, controlled layer of adhesive provided by the low melting polymer that minimizes loss of surface area of the particle.

Other types of functional particulate matter include silica, zeolite, molecular sieve, clay, alumina, ion exchange resin, organic metal catalyst, metal oxide, biocide, fungicide, and virucide. For example, a fungicidal particulate matter may be incorporated into a filter structure, such as for an automobile climate control system, to remove mildew and mildew odors from the circulated air. Biocides and virucides may be incorporated into chemical defense fabrics for protection against biological warfare. Particulate sizes may range from a small size of 1 micron for biocides and fungicides for garments and some filters to 3 to 5 mm. for other filters, and vary in shape from spheroidal beads to cylinders up to about 6 mm. in length. Activated carbon beads typically are of about 400 microns in size. The foregoing list is intended to be representative of the wide variety of functional particulate matter available, and not in limitation of the substances suitable for use in the practice of the present invention.

Turning now to a discussion of the thermally bonded fibrous structure of the invention for entrapping the particulate matter within the structure of the web, Figure 6 represents the thermally bonded web of the present invention and shows a portion of the composite manufactured fibers 60 is greatly exaggerated size being bonded at the cross over points 68. These bonds 68 provided by composite fibers are generally stronger than the composite fibers themselves. As can be seen from Figure 6, the thermally bonded web forms a cage like structure in which particulate matter may be entrapped and substantially precluded from migrating in the web. The particulate matter is dispersed into the structure of the web and then heat is applied to thermally bond the web.

Turning now to a discussion of the various processes by which the fibrous structures of the present invention can be manufactured, Figure 7 represents in highly schematic form the formation of a fibrous structure 20 as represented in Figure 1 from a single web of fibers 24 having particulate matter 30 distributed therein. Specifically, a carding machine 70 cards composite thermoplastic staple fibers 22, such as fiber 60 shown in Figure 4 having a lower melting nylon sheath and a higher melting polyester core and forms a web 24 onto an endless moving belt 72. For carding, the fibers typically will have been crimped. Particulate matter 30, such as activated carbon beads, are applied from a shaker 74 located adjacent the carding machine. The web is opened to a sufficient degree and the beads are of a sufficient size and weight to become entrapped in the interior of the web rather than remaining on top of the web. The carbon beads may be heated and applied to the web for localized meniscus bonding with the sheath of the composite fibers. Alternatively, heat may be applied, preferably infrared heat, from a heater 76 to melt the sheath and cause the carbon particles to become adhered and immobilized within the web. Heat from heater 76 also serves to thermally bond the fiber structure to entrap the particles in the web, as is illustrated in Figure 5 and 6. If desired, fibrous structure 24 may be further bonded by the application of pressure by running structure 24 through a calendar nip (not shown).

Particulate matter may be applied from one or more scatter coaters, engraved rolls, or screen conveyors. An inclined ramp may be used to spread the particulate and to enmesh the particulate within the web. The concentration of particulate can be controlled by adjusting the ramp angle. The particulate may be heated or cold. The particulate may be added in one or more layers to improve uniformity, increase bed depth, or to provide two layers of particulates each performing a different function. Distribution of particulate matter



within the web depends to some extent on the denier of the fibers used and the size and density of the particulate matter chosen.

Figure 8 represents in highly schematic form the formation of a fabric structure 32 as is illustrated in Figure 2. Carding machine 78 forms an open web 34 of composite thermoplastic fibers 40, such as sheath-core staple fibers 60, that are deposited on endless moving belt 72. Web 34 is contacted under light pressure with a calendar roll 80 to partially densify web 34. Web 34 should be densified only to the extent that numerous individual fibers still remain extending from the surfaces thereof for intermingling with the fibers of web 24.

Another carding machine 70 deposits an open web 24 of composite thermoplastic fibers 22 onto partially densified web 34. Fibers 22 may be the same or different as fibers 40 in the partially densified web, and the fibers of the two webs become substantially intermingled at the interface 36 of the webs. Fibers 22 may be sheath-core staple as shown in Figure 4. Particulate matter is applied in the same manner as is shown in Figure 7. Heat, preferably infrared heat is applied as shown in Figure 7. The heat applied also bonds the intermingled fibers 40 of the partially bonded web 34 to the fibers 22 of the open web 24 such that the webs form a unitary structure 32 and delamination at the interface of the two webs is substantially precluded. If desired, fibrous structure 32 may be further bonded by the application of pressure by running the structure through a calendar nip (not shown).

Figure 9 represents in highly schematic form the preparation of a fibrous structure 42 similar to that illustrated in Figure 3. The formation of webs 34 and deposition of web 24

thereon with particulate matter is the same as for Figure 8. However, prior to thermally bonding the structure, another carding machine 82 deposits a carded web of composite fibers 46 onto open web 24. Web 46 may comprise the same or different fibers as webs 24 or 34. For example, web 46 may comprise sheath-core fibers 60 having a lower melting nylon sheath and a higher melting polyester core as are represented in Figure 4.

Particulate matter 48 is distributed into the web 46 from a shaker 84. Particulate matter 48 may be the same as particulate matter 30 of web 24 if it is desired to provide more particulate matter surface area and greater depth of particulate matter within the structure. Also contemplated within the practice of the invention is that different particulates can be incorporated into the webs to provide a multi-functional fibrous structure. For example, particulate matter 30 may comprise activated carbon beads for the selective adsorption of gases. Particulate matter 48 may comprise a biocide. Together, these components in the structure 42 provide a defense fabric that may be suitable for use in protecting military personnel from poison gas and bacteriological warfare tactics.

Also shown in Figure 9, a fourth layer 52 may be applied to the structure 42. Layer 52 is shown being applied from a roll 86 to the surface of web 46. Layer 52 may comprise a partially densified web of fibers or a fabric of thermoplastic manufactured fibers. It should be noted that rolling a partially densified web onto a reel has the effect of further densifying the web. The entire structure 42 is subjected to heat from a heater 88, preferably infrared heat, to bond layer 52 to the structure. Finally, if desired, the entire structure 42 may be passed through a calendar nip 90 to further bond the structure.

The web for the fabric structure of the invention may be dry formed using carding machines as illustrated above. Alternatively, the web may be formed by air laying staple fibers, either crimped or not, spin bonding of continuous filaments, or wet forming, typically of non-crimped staple. Any method that produces a web having the particulate matter distributed into the interior thereof and not on the surface is suitable for practice of the invention. In each of the methods mentioned above, an underlayer or overlayer of a carded web, preformed fiber web, fabric, or membrane may be applied.

Figure 10 represents in a highly schematic form the method of wet forming a fibrous structure 91 (Figure 11) having fabric layers bonded thereto. Figure 11 represents a greatly enlarged sectional view of the fibrous structure of Figure 10. A triple headbox, represented broadly at 92, should be used to create a fibrous structure having particulate matter distributed on the interior thereof and not extending into the surfaces. First headbox 94 deposits an aqueous suspension of thermoplastic manufactured fibers 95 (Figure 11) onto an endless foraminous forming wire 96. Vacuum is applied through the forming wire to remove a portion of the liquid and form a web and has the effect of partially densifying the web. Second headbox 100 deposits an aqueous suspension of composite thermoplastic fibers 101 and particulate matter 102 (Figure 11) onto the web from the first headbox. These fibers should have a lower melting component and a higher melting component, such as, for example, fibers 60 illustrated in Figure 4. Vacuum continues to be applied through the forming wire to remove the aqueous component of the suspension of thermoplastic fibers 104 (Figure 11) and deposits this suspension. Third headbox 103 supplies an aqueous suspension of thermoplastic fibers 104 (Figure 11) and deposits this suspension onto the web formed from the second headbox. The fibers supplied from the first and third headboxes 94 and

103, respectively, may be the same or different from the fibers 101 deposited from the second headbox 100.

One of the benefits of wet forming the fabric structure of the invention from a triple headbox as shown at 92 is that the fibers of the webs from each of the headboxes become well intermingled as the aqueous component is removed under vacuum through the forming wire. The triple headbox wet forming process results in a fabric structure that is unitary and not substantially subject to delamination of the particulate containing layers.

Also as shown in Figure 10, fabrics 105 and 106, which comprise manufactured fibers, may be applied to each side of the fabric structure from rolls 108 and 110, respectively. Heat is supplied from heater 112, preferably infrared heat, to thermally bond the web structure, entrapping the particulate matter in the structure, and to fuse the particulate matter to the individual fibers in the structure. Through-air heat is supplied from heater 114 to complete drying of the structure.

Notwithstanding the foregoing description, it is also set forth that the fibrous structure as set forth may be used in adsorptive media technology. When used, the media may take various forms including pleated, tubes, pockets (as it pocket filters), blankets, rolls, bags covering wallcovering and in ducts and ductliners, and duct form itself. The media can be used singly or in combination with other fabrics, filter media, films, plastics and membranes. Numerous end use applications exist for the fibrous structure in the filter media technology. Such uses include interior passenger compartments in cabin air filters suitable for trains, subways, buses, airplanes, construction vehicles, agricultural vehicles, submarines, landfill

equipment and garbage/refuge control. Other uses exist for the heating, ventilation, air conditioning uses in residential air purifiers, clean room, pharmaceutical processing, food processing, smoking lounges, funeral homes, etc. Also, the media may be used in equipment such as office equipment, casket liners, bandages, packaging and protective wrapping. Numerous military civil defense and police uses exist for chemical and underground bunkers. Environmental uses include solving emission control, chemical waste cleanup, recycled solvents, incineration, sewage plants, sterilization equipment. Also the media may be used in liquid filtration such as high purity water and color or byproduct removal in such operations as producing whisky, vinegar, etc.

While the invention has been described with reference to specific preferred embodiments, it should be understood that the invention is not intended to be limited to the embodiments illustrated in the drawings. On the contrary, the invention includes all alternatives, modifications, and equivalents that may be included within the scope and spirit of the invention as defined by the appended claims.

## THAT WHICH IS CLAIMED IS:

1. A fibrous structure comprising a nonwoven web of manufactured fibers, said web having upper and lower surfaces, and particulate matter immobilized within said web without substantially extending into said upper and lower surfaces.
2. The fibrous structure of Claim 1 wherein said structure is thermally bonded.
3. The fibrous structure of Claim 1 wherein said structure is pressure bonded.
4. The fibrous structure of Claim 1 wherein said nonwoven web of manufactured fibers included thermoplastic manufactured fibers and particulate matter is immobilized within said nonwoven web by fusion bonding of said particulate matter to said thermoplastic manufactured fibers.
5. The fibrous structure of Claim 1 wherein said manufactured fibers include composite fibers selected from among composite continuous thermoplastic filaments, composite thermoplastic staple, and blends thereof, said filaments and staple having a relatively lower melting component and a relatively higher melting component, and wherein said particulate matter is fusion bonded to said lower melting component.

6. The fibrous structure of Claim 5 wherein said composite fibers are selected form among concentric and eccentric sheath-core fibers, side-by-side fibers, and blends thereof and said fibers are bonded at the cross over points.

7. The fibrous structure of Claim 6 wherein said lower and higher melting components differ in melting point by at least about 20°C.

8. The fibrous structure of Claim 6 wherein said lower melting component is nylon and said higher melting component is polyester.

9. The fibrous structure of Claim 5 wherein said manufactured fibers include fibers of a single polymer in an amount of from 10% to 80% by weight of the structure.

10. The fibrous structure of Claim 5 further comprising cellulose acetate fibrets.

11. The fibrous structure of Claim 1 wherein said manufactured fibers include structural fibers of denier from about 6 dpf to about 10,000 dpf.

12. The fibrous structure of Claim 1 wherein said manufactured fibers include fibers of microdenier of from about 0.1 dpf to about 1.0 dpf.

13. The fibrous structure of Claim 1 wherein said particulate matter is selected from among activated carbon, silica, zeolite, molecular sieve, clay, alumina, ion exchange resin, organic metal catalyst, metal oxide, biocide, fungicide, and virucide.

14. The fibrous structure of Claim 1 further comprising a partially bonded nonwoven web of manufactured fibers bonded to one of said upper and lower surfaces of said web of Claim 1.

15. The fibrous structure of Claim 1 further comprising one or more fabric layers selected from among the group of knit, woven, and nonwoven fabrics bonded to said web.

16. A fibrous structure comprising a core of a nonwoven web of thermoplastic manufactured composite fibers, said composite fibers having a denier of from about 1 dpf to about 15 dpf and comprising a relatively higher melting component and a relatively lower melting component and being thermally bonded at the cross over points, said web having upper and lower surfaces, and particulate matter fusion bonded to said relatively lower melting component within said web without substantially extending into said upper and lower surfaces.

17. The fibrous structure of Claim 16 wherein said composite fibers are selected from among the group consisting of sheath-core fibers having a relatively lower melting sheath component and a relatively higher melting core component and side-by-side



bi-component fibers, and wherein in cross section each component comprises about one half the area of the fiber.

18. The fibrous structure of Claim 17 wherein said composite fibers are concentric sheath-core staple fibers having a nylon sheath and a polyester core, said nylon sheath having a melting point of from about 175°C to about 185°C, and said polyester core having a melting point of about 240°C to 256°C.

19. The fibrous structure of Claim 16 wherein said core further comprises from about 10% to about 80% by weight of non-composite thermoplastic manufactured fibers.

20. The fibrous structure of Claim 19 wherein said thermoplastic fibers are a microdenier staple having a denier in the range of from about 0.1 denier per filament to about 1.0 denier per filament.

21. The fibrous structure of Claim 16 wherein said particulate matter is selected from among activated carbon, silica, zeolite, molecular sieve, clay, alumina, ion exchange resin, organic metal catalyst, metal oxide, biocide, fungicide, and virucide.

22. The fibrous structure of Claim 21 wherein said particulate matter comprises particles having a nominal particle diameter of from about 0.1 microns to about 5 mm.

23. The fibrous structure of Claim 21 wherein said particulate matter is activated carbon having a nominal particle diameter of about 400 microns and is present in the fibrous structure in an amount by weight of about 132 g/m<sup>2</sup>.

24. A fibrous structure comprising:

(a) a core of a nonwoven web of thermoplastic manufactured composite fibers, said composite fibers comprising a relatively higher melting component and a relatively lower melting component, said web having upper and lower surfaces;

(b) particulate matter fusion bonded to said relatively lower melting component within said core web; and

(c) a partially bonded nonwoven web of thermoplastic manufactured composite fibers, said composite fibers comprising a relatively higher melting component and a relatively lower melting component and being thermally bonded at the cross over points, said partially bonded web being bonded to said core web on one of said surfaces of said core web.

25. The fibrous structure of Claim 24 further comprising an additional web bonded to said core web opposite said web of element (c).

26. The fibrous structure of Claim 25 wherein said additional web comprises a fabric selected from among the group consisting of woven, knit, and nonwoven fabrics.

27. The fibrous structure of Claim 25 wherein said additional web comprises a partially bonded nonwoven web of thermoplastic manufactured composite fibers, said composite fibers comprising a relatively higher melting component and a relatively lower melting component and being thermally bonded at the cross over points.

28. A process for making a fibrous structure comprising the steps of:

(a) forming an open fibrous core web of entangled thermoplastic manufactured fibers that contains particulate matter distributed within the web between the surfaces of the web without substantially extending into the web surfaces;

(b) immobilizing the particulate matter within the interior of the web; and

(c) bonding the thermoplastic fibers.

29. The process of Claim 28 wherein the step of forming the open fibrous web having particulate matter distributed there within is selected from among the group consisting of the steps of:

(a) forming a carded web and distributing particulate matter into the interior of the web;

(b) air forming a web and distributing particulate matter into the interior of the web;

(c) wet laying a web from an aqueous suspension of thermoplastic manufactured fibers and particulate matter between two other wet laid webs not containing particulate matter; and

(d) spinning continuous filaments; forming a web therefrom, and distributing particulate matter into the interior of the web.

30. The process of Claim 28 wherein the step of immobilizing the particulate matter within the interior of the web comprises entrapping the particles within the entangled manufactured fibers of the fibrous structure of the web and applying heat sufficient to fuse the particulate matter to the fibers.

31. The process of Claim 30 wherein heat is applied to fuse the particulate matter to the fibers by heating the web.

32. The process of Claim 30 wherein heat is applied to fuse the particulate matter to the fibers by heating the particulate matter prior to distributing the particulate matter into the interior of the web.

33. The process of Claim 28 further comprising the steps of forming additional fibrous structures containing immobilized particulate matter and bonding together the fibrous structures to form a multiple layer core fabric structure.

34. The process of Claim 28 further comprising the step of bonding at least one partially densified web to the core web.

35. The process of Claim 28 further comprising the step of bonding at least one fabric to the core web selected from among the group consisting of woven, knit, and nonwoven fabrics.

36. A process for forming a fibrous structure comprising the steps of:

- (a) forming a web comprising composite staple fibers having a lower melting component and a higher melting component;
- (b) distributing particulate matter into the web;
- (c) entrapping the particulate matter within the structure of the web and without extending substantially into the web surfaces;
- (d) applying heat to fuse the particulate matter to the fibers; and
- (e) applying heat to bond the fibers at the cross over points.

37. The process of Claim 36 wherein the step of (d) applying heat to fuse the particulate matter to the fibers comprises heating the particulate matter above the melting point of the lower melting component prior to distributing the particulate matter into the web.

38. The process of Claim 36 further comprising the steps of:

- (a) forming the web of step (a) on a partially densified web of composite thermoplastic fibers prior to distributing particulate matter into the web of step (a); and
- (b) bonding the web of step (a) to the partially densified web.

39. The process of Claim 36 further comprising the step of bonding a fabric selected from among the group consisting of woven, knit, and nonwoven fabrics to the web.

40. A process for making a fibrous structure comprising the steps of:

- (a) partially bonding a first fibrous web comprising composite manufactured fibers having one component with a melting point at least 20°C below the melting point of the other component;
- (b) forming a second fibrous web onto said first fibrous web, said second fibrous web also comprising composite fibers having one component with a melting point at least 20°C below the melting point of the other component;
- (c) dispersing particulate matter into the second web;
- (d) entrapping the particulate matter within the fibrous structure of the second web;
- (e) applying heat to fuse the particulate matter to the fibers; and
- (f) applying heat to thermally bond the fibers in the first and second webs and to thermally bond the first web to the second.

41. The process of Claim 40 further comprising the step of applying pressure to the fibrous structure to further bond the structure.

42. A process for wet forming a fabric structure having particulate matter immobilized therewithin so as substantially not to extend into the upper and lower surfaces of said structure, said process comprising the steps of:

- (a) depositing a liquid suspension of staple thermoplastic manufactured fibers onto a foraminous forming wire;
- (b) drawing vacuum to the forming wire to partially remove the liquid component and form a first web;

(c) depositing a liquid suspension of composite thermoplastic staple and particulate matter onto said first web, said staple having a lower melting component and a higher melting component;

(d) continuing to draw vacuum to form a second web;

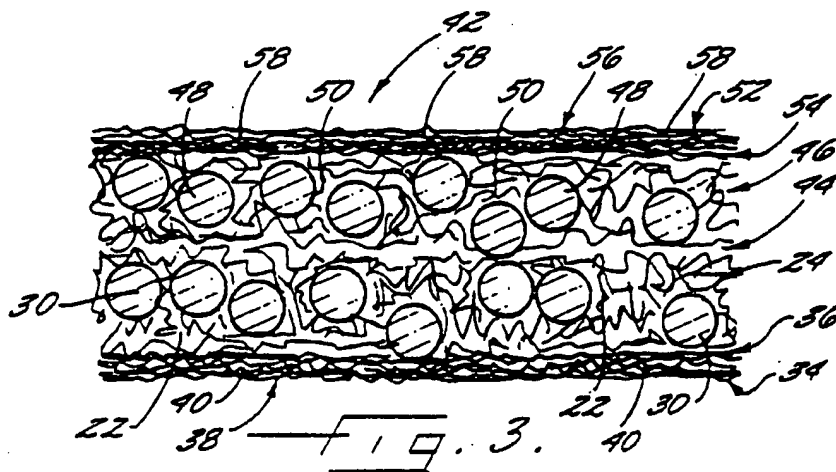
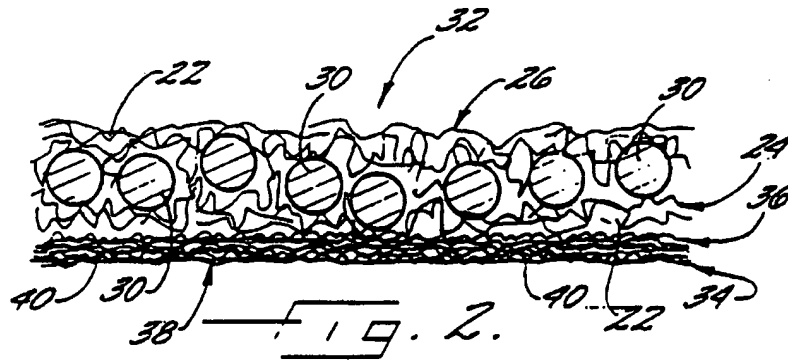
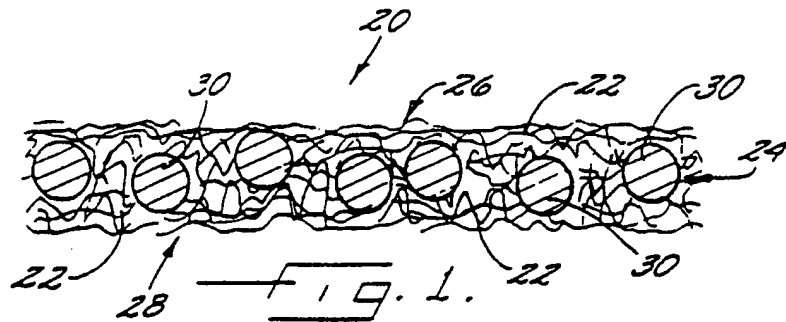
(e) depositing a liquid suspension of thermoplastic manufactured staple onto said second web;

(f) continuing to draw vacuum to obtain a third web;

(g) applying heat to thermally bond each of the said webs to form a unitary structure that substantially will not delaminate at the interface of the surfaces of the webs; and

(h) applying heat to fuse the particles to the lower melting component of the staple in the second web.

43. The process of Claim 42 further comprising the steps of applying one or more fabric layers to said structure.



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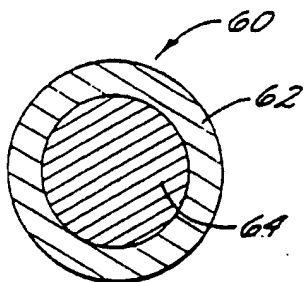


FIG. 4.

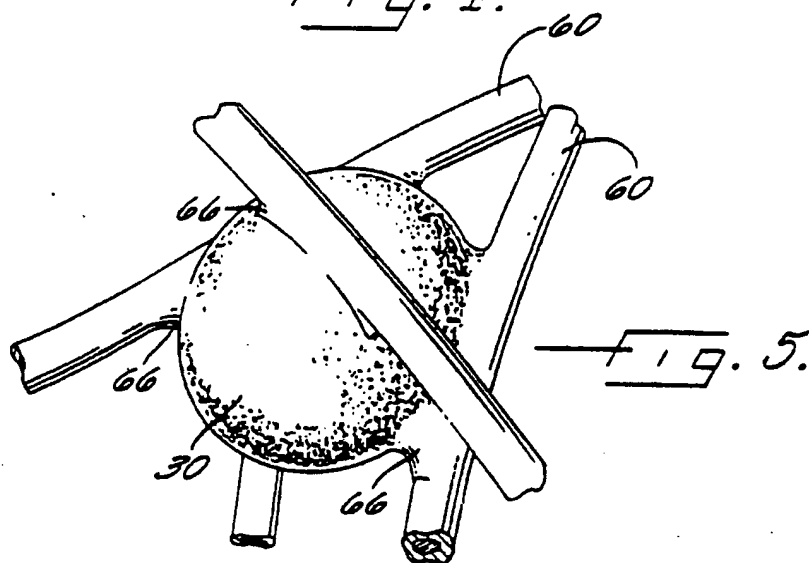


FIG. 5.

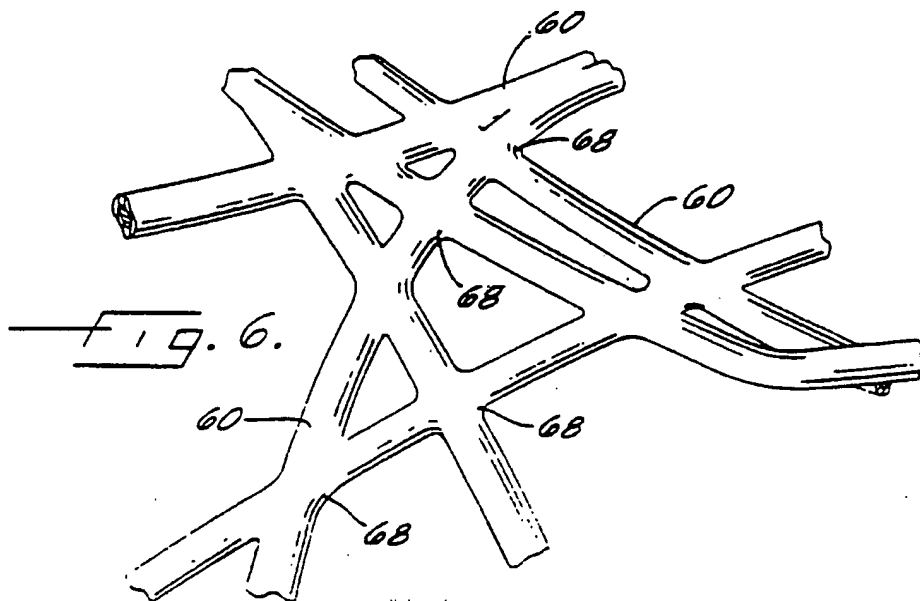
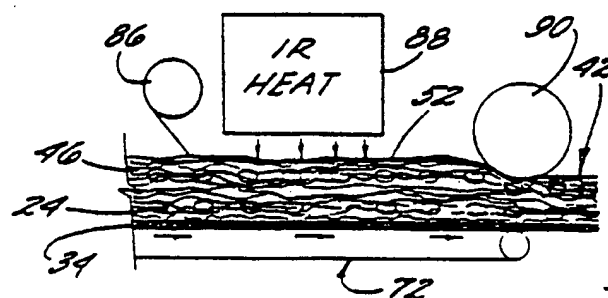
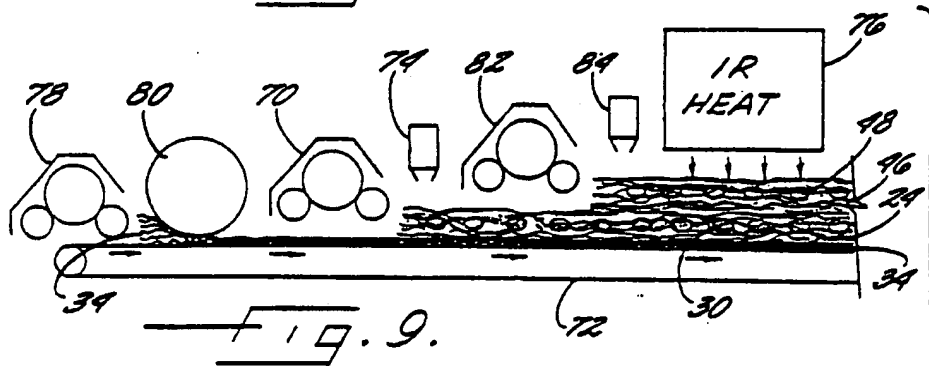
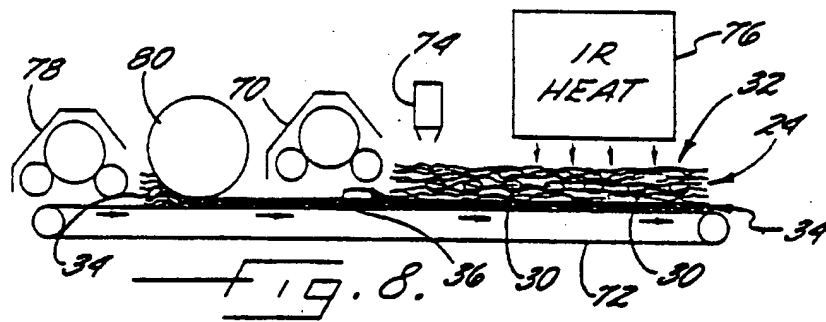
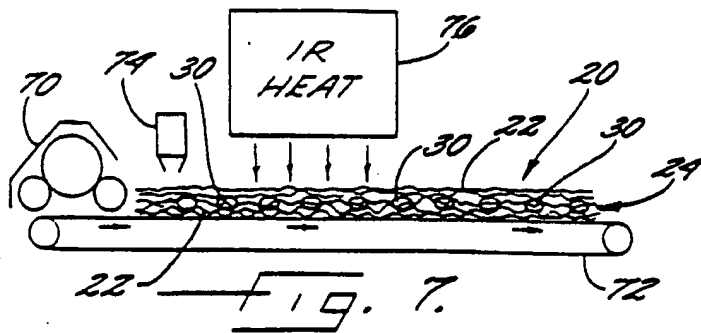
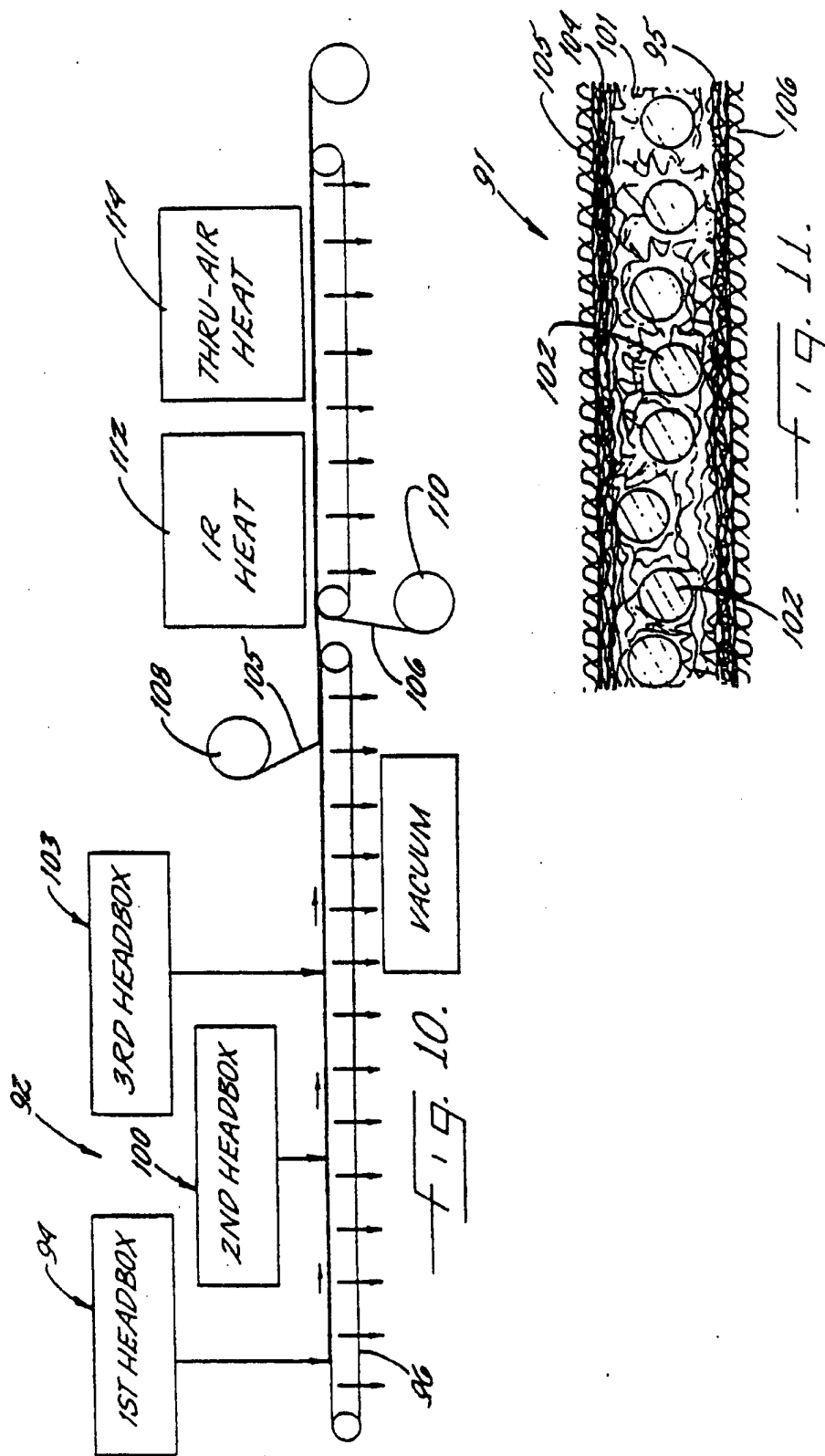


FIG. 6.

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## INTERNATIONAL SEARCH REPORT

Intern. Appl. No.

PCT/US 93/11062

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 5 D04H1/54 D04H13/00

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 5 D04H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP,A,0 257 868 (MINNESOTA MINING AND MANUFACTURING COMPANY) 2 March 1988	1-6, 9, 12-17, 19-22, 28, 30, 31, 35
A	see column 2, line 13 - column 8, line 58	23, 24, 36, 37, 40, 41
X	US,A,4 429 001 (KOLPIN ET AL.) 31 January 1984	1-4, 11, 12
A	see column 3, line 32 - column 5, line 61	9, 15
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Date of the actual completion of the international search

23 March 1994

Date of mailing of the international search report

06.04.94

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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P,X	DATABASE WPI Section Ch, Week 9326, Derwent Publications Ltd., London, GB; Class A88, AN 93-208373 & JP,A,5 131 136 (KURARAY CHEM CO LTD) 28 May 1993 see abstract -----	1,2,4-6, 13,15
A	EP,A,0 275 047 (KANEBO LTD.) 20 July 1988  see page 3, line 3 - page 7, line 20 -----	1,2,4-8, 13

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 93/11062

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A-0257868	02-03-88	US-A- 4868032	19-09-89
		US-A- 4681801	21-07-87
		DE-D- 3787775	18-11-93
		ES-T- 2044939	16-01-94
		JP-A- 63059425	15-03-88
-----			
US-A-4429001	31-01-84	AU-B- 561405	07-05-87
		AU-A- 1203183	08-09-83
		AU-B- 576698	01-09-88
		AU-A- 7472787	15-10-87
		EP-A,B 0088533	14-09-83
		JP-C- 1645299	28-02-92
		JP-A- 58163438	28-09-83
JP-B- 63066569	21-12-88		
-----			
EP-A-0275047	20-07-88	JP-A- 63175117	19-07-88
		DE-A- 3869967	21-05-92
		US-A- 5064599	12-11-91
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